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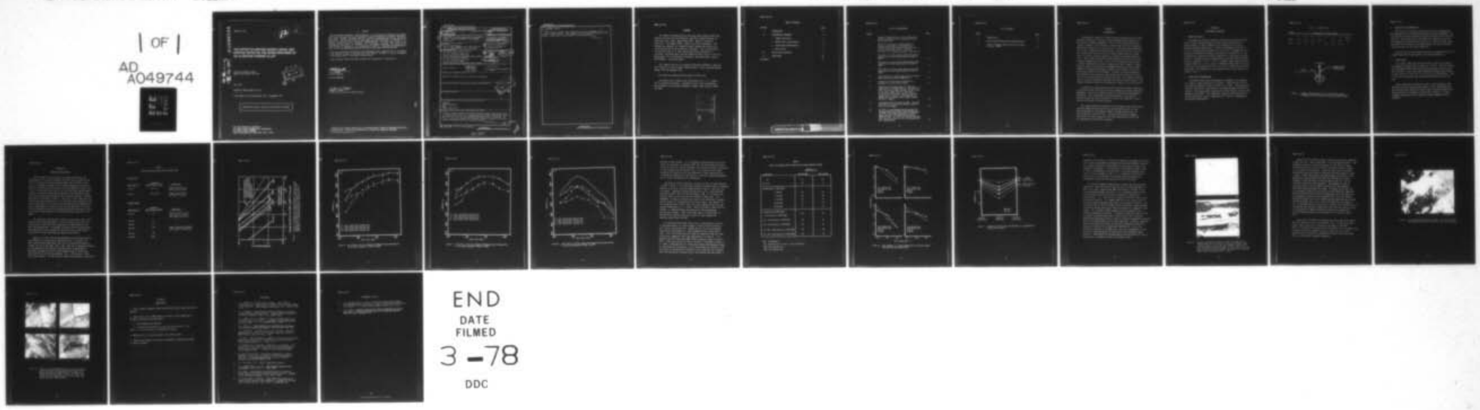

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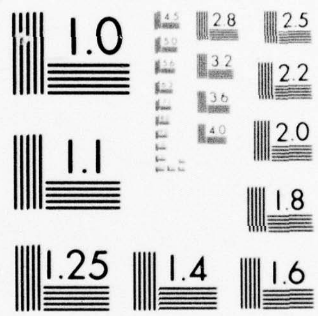

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THE EFFECT OF VARYING QUENCH RATES AND HEATING RATES ON THE AGING RESPONSE OF AN ALUMINUM POWDER ALLOY

Structural Metals Branch
Metals and Ceramics Division

July 1977

TECHNICAL REPORT AFML-TR-77-67

Final Report for Period October 1975 - September 1976

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L R Bidwell

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Project Engineer

FOR THE DIRECTOR

Nathan G. Tupper

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Acting Chief, Structural Metals Branch

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: AFML-TR-77-67
2. GOVT ACCESSION NO.:
3. RECIPIENT'S CATALOG NUMBER:
4. TITLE (and Subtitle): THE EFFECT OF VARYING QUENCH RATES AND HEATING RATES ON THE AGING RESPONSE OF AN ALUMINUM POWDER ALLOY.
5. TYPE OF REPORT & PERIOD COVERED: Final Technical Report, OCT 1975 – SEP 1976.
6. PERFORMING ORG. REPORT NUMBER:
7. AUTHOR(s): Walter M. Griffith, Air Force Materials Lab; Marlin M. Cook, University of Cincinnati
8. CONTRACT OR GRANT NUMBER(s): F33615-76-C-5227 new
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Structural Metals Branch (LLS), Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433
10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 24180204, 73510175; 02301
11. CONTROLLING OFFICE NAME AND ADDRESS: Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio 45433
12. REPORT DATE: July 1977
13. NUMBER OF PAGES: 30
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): Same as Block 11 — 30p.
15. SECURITY CLASS. (of this report): Unclassified
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE:
16. DISTRIBUTION STATEMENT (of this Report): Approved for public release; distribution unlimited.
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report):
18. SUPPLEMENTARY NOTES:

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Aluminum
Powder Metallurgy
MA87
Quench Sensitivity

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This research examines the effect of varying quench rates and the effect of varying heating rates on the aging response of powder aluminum MA87 alloy. The effect of a six-day room temperature pre-age was also considered. The results obtained are compared with results of similar research on 7075 ingot product and supplemented with electron microscopy.

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20. ABSTRACT (Cont'd)

The results indicate little difference in terms of hardness, in the response of MA87 and 7075. This suggests that the quench sensitivity of both alloys is similar and is attributable to the same mechanism.

FOREWORD

This report was prepared by the Structural Metals Branch, Metals and Ceramics Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433, under Project 2418, "Metallic Structural Materials," Task Number 241802, "Metals and Alloys Technology." The research was conducted on a cooperative basis between the University of Cincinnati and the Air Force Materials Laboratory. The co-investigators for the effort were M. M. Cook, Research Assistant, University of Cincinnati, who worked under Contract F33615-76-C-5227; and W. M. Griffith, Metallurgist, Air Force Materials Laboratory, who worked under in-house WUD 24180204, "Structural Metals."

This research was part of a program investigating methods of improving the reliability of high strength aluminum alloys. It was conducted between October 1975 and September 1976.

This report was submitted by the authors in March 1977.

The authors wish to express their gratitude to Dr. T. M. F. Ronald for his technical assistance. The material used in this investigation was purchased from the Aluminum Company of America under Contract F33615-74-C-5077.

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SECTION I

INTRODUCTION

The mechanical properties of heat-treatable aluminum alloys are often adversely affected by slow cooling rates developed during quenching from the solution treatment temperature. This so-called quench sensitivity creates a problem in thick sections of these alloys since their midplanes experience a relatively slow cooling rate. It has been found (References 1 and 2) that in 7000-series aluminum alloys a slow cooling rate allows time for precipitation directly from solid solution because the solute atoms (Mg, Zn, Cu) have sufficient mobility to migrate to favorable nucleation sites. These sites include primary grain boundaries and relatively large insoluble dispersoid particles. Due to this heterogeneous precipitation a uniform distribution of solute no longer exists and, further, a reduced amount of solute is available for strengthening during artificial aging. Attempts to overcome the problems created by slow internal cooling rates often create equally serious problems. Increases in product complexity and thickness lead to distortion and complex residual stresses if cooling rates fast enough to maintain properties are used. Thus, quench sensitivity can be a serious problem affecting both alloy choice and part design if the highest attainable properties and dimensional stability are to be assured.

In addition to rapid cooling rates from solution treatment temperature, a slow heating rate to the artificial aging temperature has been identified (References 1 and 3)as an important factor in attaining maximum mechanical properties. This is explained by noting that there is more time for Guinier-Preston (GP) zones to grow to a critical size resulting in a more copious distribution of stable GP zones.

This research examines the effect of varying quench rates from solution treatment temperature and the effect of varying heating rates to final aging temperature on the hardness of MA87, a powder aluminum alloy of current interest to the Air Force. The results obtained are compared with results of similar research on the ingot metallurgy alloy 7075 and are supplemented with electron microscopy.

SECTION II

EXPERIMENTAL PROCEDURE

1. POWDER PROCESSING

The powder was produced, processed, and forged at the Alcoa Technical Center, and was obtained through Air Force Contract F33615-74-5077. Complete details of powder preparation and subsequent processing are presented elsewhere (Reference 4). Briefly, the powder was atomized in air to an average particle diameter of 13.2 microns. The powder was cold pressed to 70% theoretical density and placed in a cylindrical can. The pre-compaction treatment consisted of evacuation to 35μm Hg while heating to 970°F. The compaction was accomplished by blind die extrusion at 900°F and 90 ksi pressure. A 12 in. x 7.25 in. diameter compact was then upset forged at 700°F to a 2.4 in. x 16.0 in. diameter pancake. Radial slices 0.5 inch-thick were sectioned into 0.25 x 0.5 x 0.5 in. coupons for use in the experiments. The compositions of the materials used in this research are listed in Table 1.

2. QUENCH RATE DETERMINATIONS

A shielded chromel-alumel thermocouple was embedded in the midplane of a coupon as shown schematically in Figure 1. After heating to 910°F the coupon was immersed in water at either 72° or 210°F. The thermocouple output was measured by a high response, multispeed Honeywell Electronic 19 recorder. The average quench rates were calculated from the cooling curves between 750° and 550°F. This temperature range was chosen because slow cooling through this range of temperatures will result in heterogeneous precipitation (Reference 2).

TABLE 1. COMPOSITIONS

ALLOY	COMPOSITION - WEIGHT PERCENT							
	Cu	Mg	Zn	Co	Cr	Si	Fe	Oxygen
MA87	1.55	2.35	6.41	0.39	-	0.03	0.05	0.39
7075	1.39	2.52	5.75	--	0.19	0.17	0.16	-

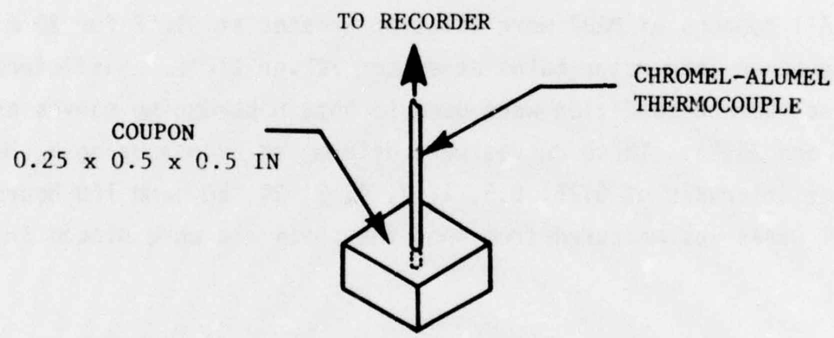


Figure 1. Schematic Representation of the Thermocouple-Coupon Arrangement Used to Determine Cooling and Heating Rates

3. HEATING RATE DETERMINATIONS

Heating rates to be used to attain artificial aging temperatures were measured by the same thermocouple-coupon-recorder arrangement. The slow heating rates were obtained by placing the coupon into an initially cold oven. Since heating rates were found to be affected by the final aging temperature, adjustments were made in air intake and exhaust ports to control air circulation so that approximately the same slow heating rate was obtained for each aging temperature.

Fast heating rates were measured by placing the sample directly into a Wood's Metal bath already heated to the desired temperature.

4. AGING CURVES

All coupons of MA87 were solution treated at 910°F for 20 minutes and quenched into water baths at either 72° or 210°F. Sufficient coupons for each quench condition were used to obtain hardening curves at 250°, 300°, and 350°F. These curves were determined by measuring R_B hardness at time intervals of 0.25, 0.5, 1, 2, 4, 8, 24, 50, and 150 hours. Time in all cases was measured from when the specimens were placed in the oven.

Several additional specimens were held at room temperature after quenching to monitor natural aging response. These room temperature aged specimens were subsequently aged at 250°F to determine what effect natural aging has on response to further heat treatment. All other specimens were placed into the aging environment immediately after quenching.

SECTION III

RESULTS AND DISCUSSION

Table 2 summarizes the results of the quench and heating rate determinations. For samples quenched into 210°F water average cooling rates of 41°and 46°F/sec were measured between 750° and 550°F in two trials. For samples quenched into 72°F water average cooling rates of 1666° and 1819°F/sec were measured for the same temperature range in two trials. In both cases these results for 0.25 inch-thick coupons compare favorably with those of Hunsicker (Reference 5) who determined cooling rates for a number of quench mediums using different thickness specimens. These data are included in Figure 2. Also, note in this figure that the slow cooling rates measured for a 0.25 inch-thick coupon quenched in 210°F water is equivalent to the cooling rate experienced by the midplane of a section approximately 3 inch-thick that has been quenched in 72°F water. Thus, the hardness data obtained represents what one would expect on the surface and at the midplane of a 3-inch-thick product quenched in 75°F water.

The aging ovens were adjusted to obtain a slow heating rate of ∿5°F/minute for each aging temperature. The fast heating rates varied from 370°F/minute for the 250°F aging temperature to 1080°F/minute for the 350°F aging temperature. No attempts to equalize the fast heating rates were made because it was assumed that the variations at these heating rates would not affect age-hardening kinetics significantly.

Figures 3, 4, and 5 show the age-hardening curves developed from all the combinations of quench rates and heating rates. The results for 250° and 300°F aging temperatures show that the greatest differences in hardness are due to the quench rates. Note, that the as-quenched difference of 6 R_B between the slow and fast quenched coupons is approximately maintained throughout the entire aging sequence except at short times where heating rate effects are expected. This is not the case for the 350°F aging sequence. At 350°F the fast heating rate exhibits a significant

TABLE 2

QUENCH RATE AND HEATING RATE DETERMINATIONS

QUENCH RATES

TEMP RANGE °F	MEASURED QUENCH RATE °F/SEC	CONDITION
750-550	41, 46	Sample quenched into 210°F H_2O (2 trials)
750-550	1666, 1819	Sample quenched into 72°F H_2O (2 trials)

HEATING RATES

TEMP RANGE °F	MEASURED HEATING RATE. °F/MIN	CONDITION
100-240	4.8	Sample started in cold oven with air intake and exhaust ports open
100-295	5.6	"
100-345	5.2	"
100-230	370	Sample immersed in Wood's Metal bath at temperature
100-285	840	"
100-340	1080	"

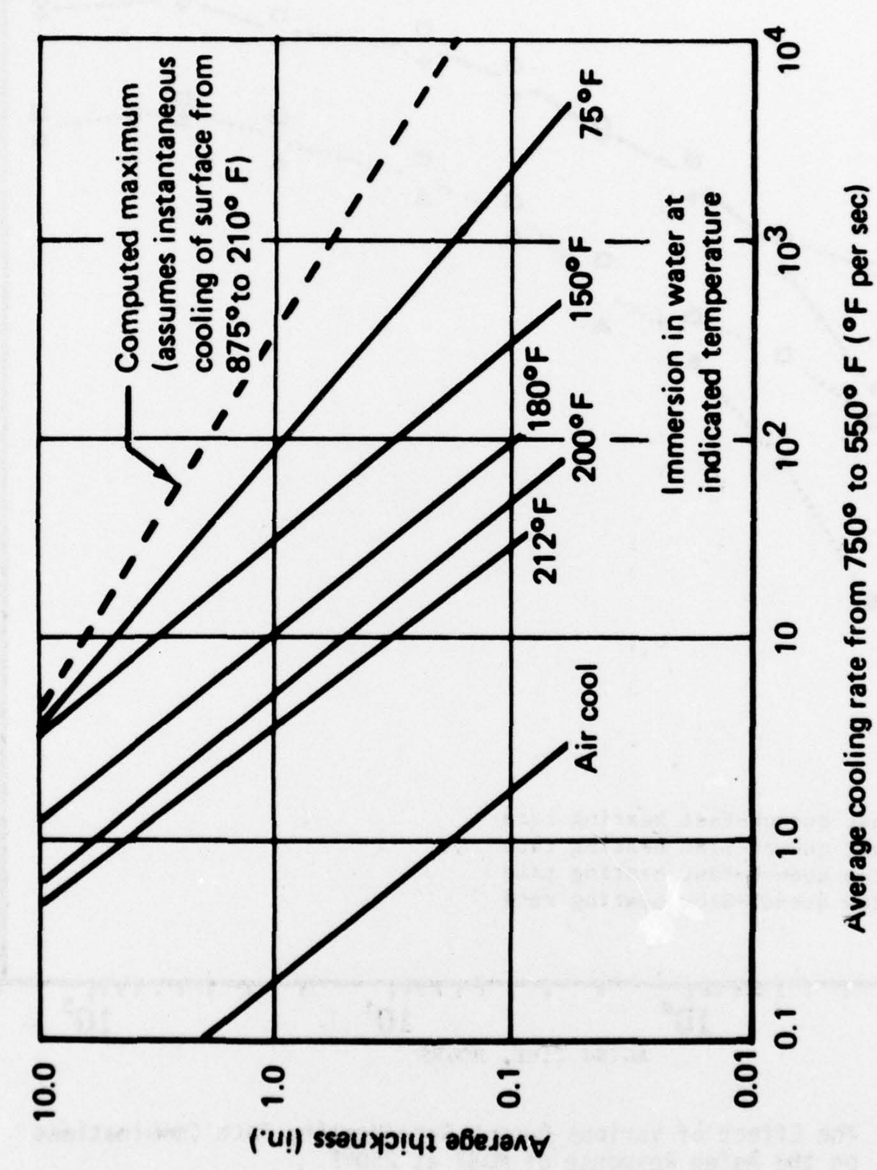


Figure 2. Effects of Thickness and Quench Medium on Average Cooling Rates at the Midplane of Aluminum Alloy Sheet and Plate Quenched from Solution Temperature (after, Hunsicker, Reference 5, as reported by Hyatt, Reference 14)

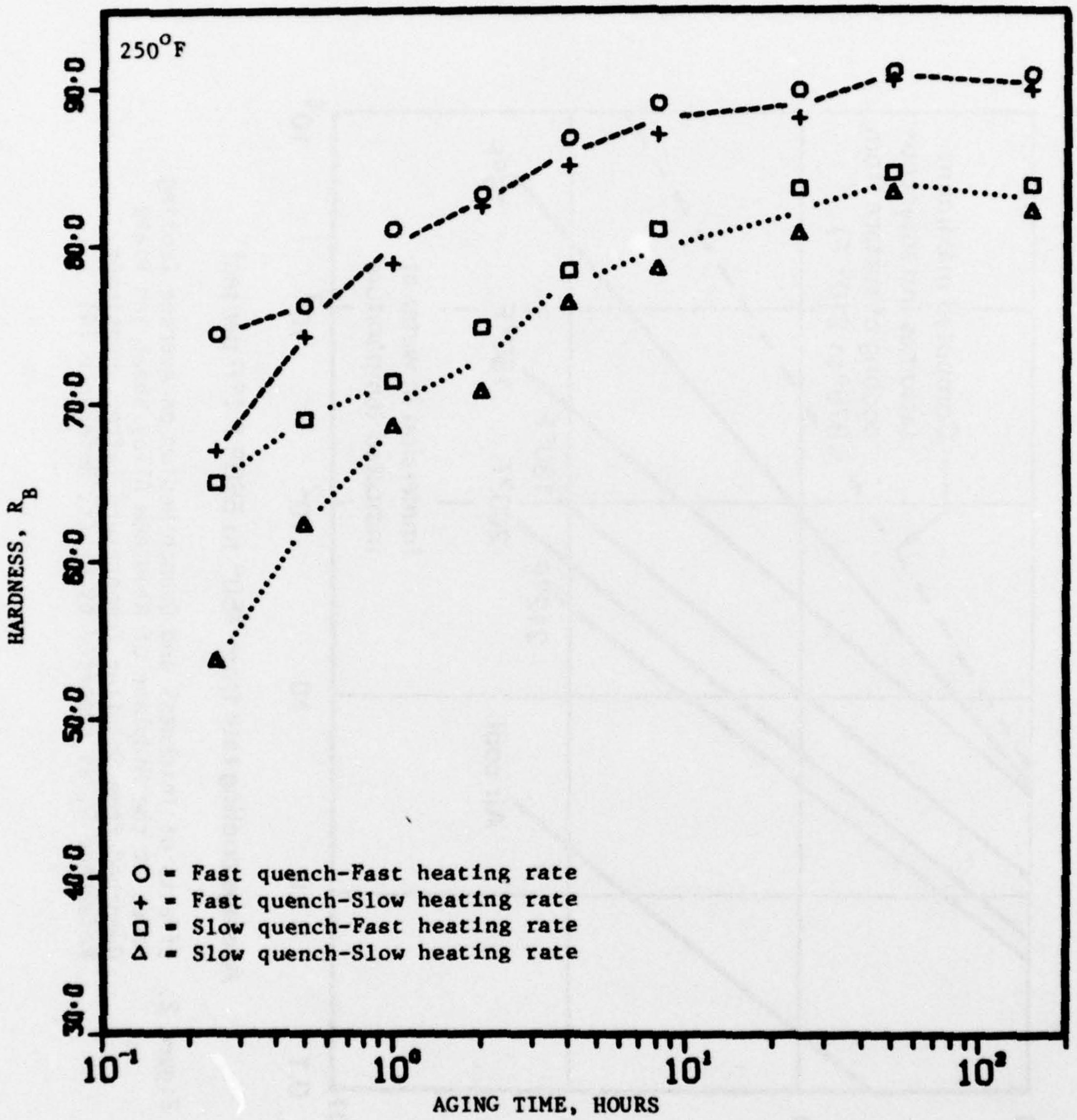


Figure 3. The Effect of Various Quench Rate/Heating Rate Combinations on the Aging Response of MA87 at 250°F

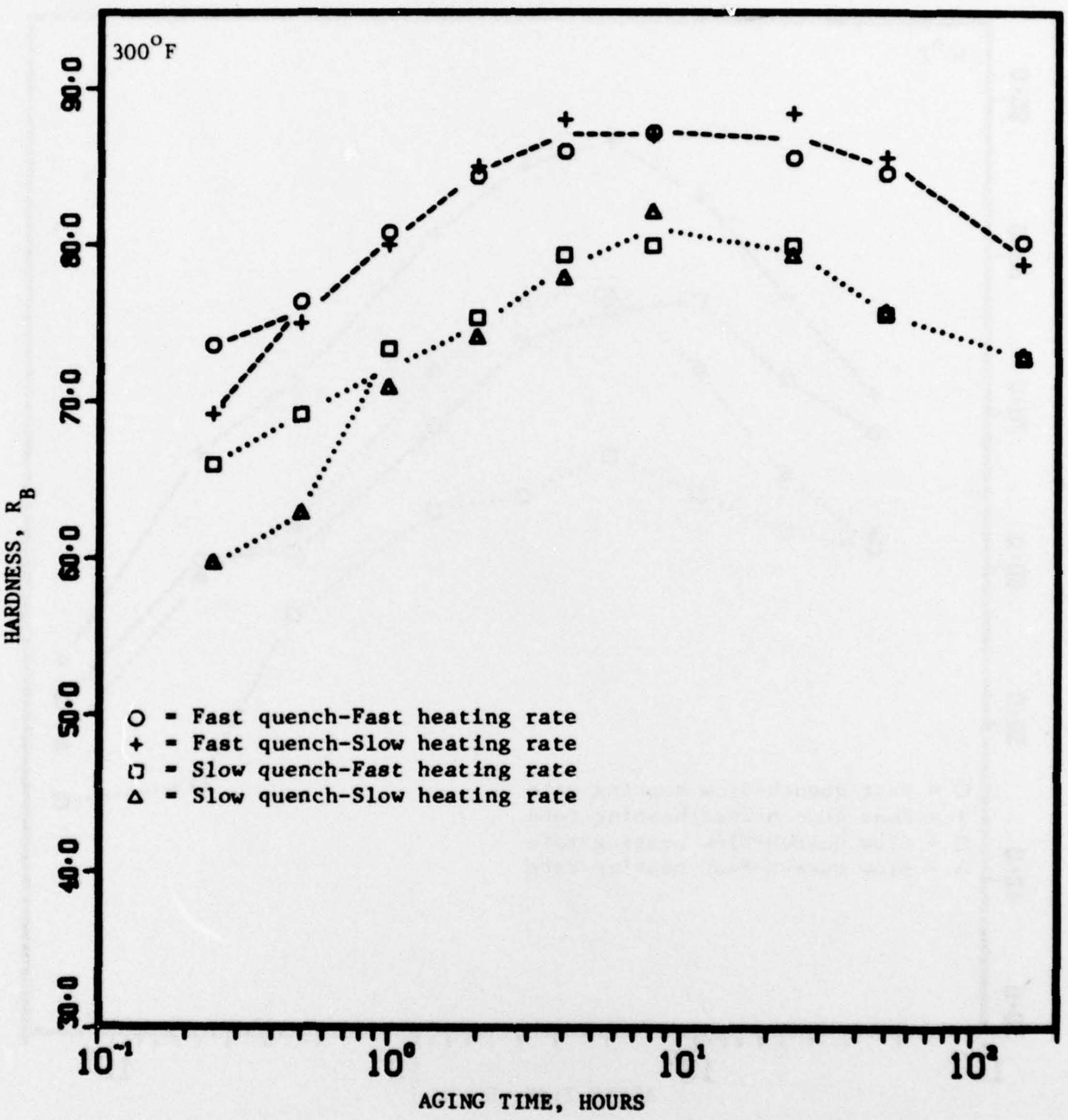


Figure 4. The Effect of Various Quench Rate/Heating Rate Combinations on the Aging Response of MA87 at 300°F

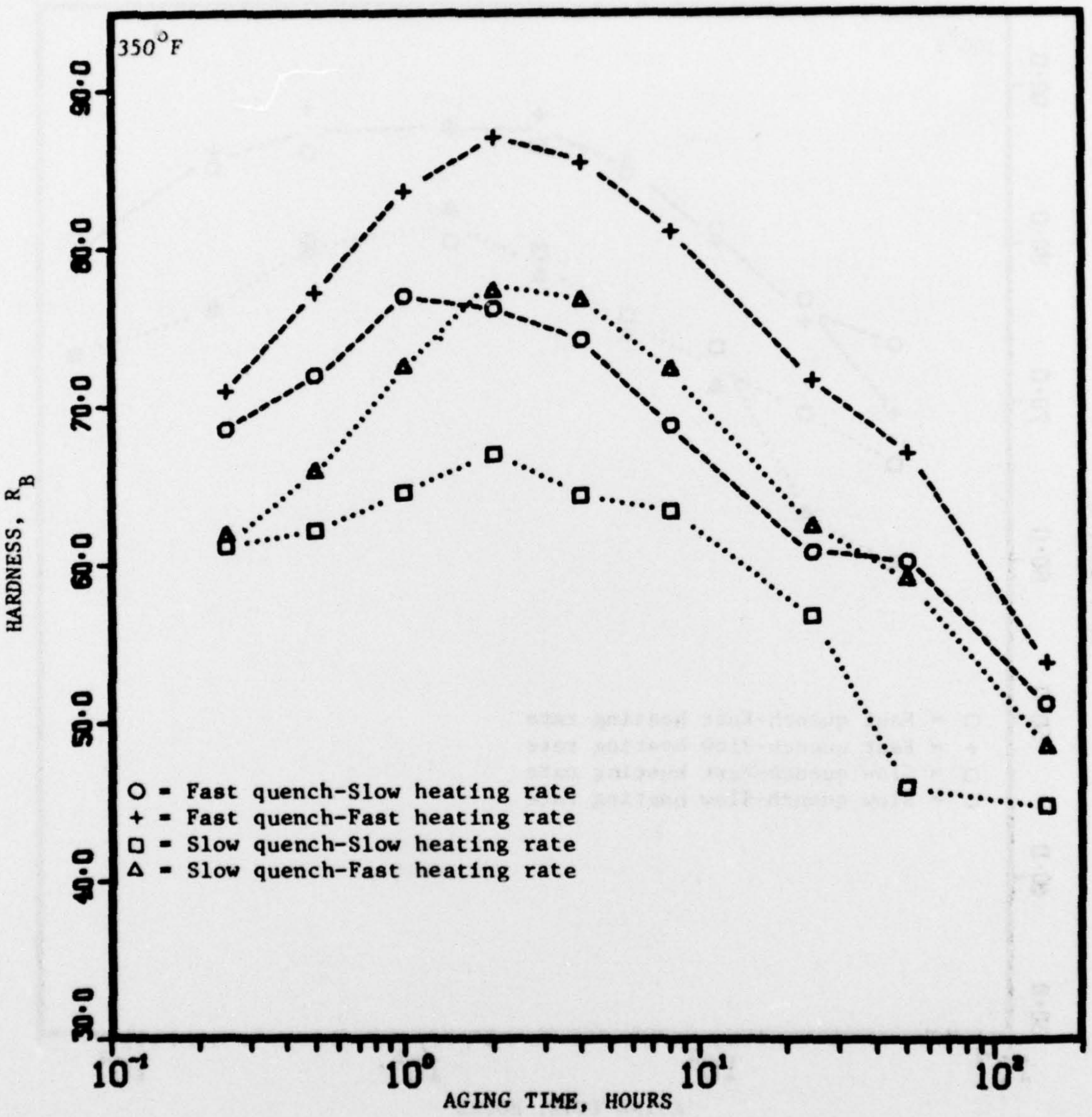


Figure 5. The Effect of Various Quench Rate/Heating Rate Combinations on the Aging Response of MA87 at 350°F

decrease in peak hardness. It is reasonable to believe that the critical radius of a GP zone is so large at 350°F that the fast heating rate does not allow sufficient time for formation of a distribution that is equivalent to that formed by the slow heating rate. This larger critical radius precludes the fineness of the zones developed at lower temperatures and is therefore responsible for the decrease in relative hardness experienced for all quench rate/heating rate combinations.

The effect of natural aging for six days on artificial aging response is shown in Table 3. The fast quench specimens increased from an as-quenched hardness of R_B 42 to a naturally-aged hardness of R_B 78. Similarly, the slow quench specimens increased from R_B 48 to R_B 72. These specimens were then heated to 250°F at the fast heating rate. As is evident from the data, there is an initial drop in hardness in both cases. This phenomenon is known as reversion and is due to the resolution of unstable solute clusters at the beginning of artificial aging. This behavior is not unexpected since similar results for other aluminum alloys have been reported (References 5, 6, 7, and 8). After 30 minutes at 250°F the lost hardness is regained because precipitation of the age-hardening precipitate has begun. Table 3 also shows there is little effect of heating rate or natural aging for six days on final hardness for specimens aged 24 hours at 250°F.

For aluminum alloys quench sensitivity is often defined as the loss in peak aged hardness (or yield strength, stress corrosion resistance, etc.) due to slow quench rates. Quench sensitivity can be determined from plots of peak hardness vs. aging temperature for various quench rates. Plots of the data from this study are compared to data for plate rolled from a commercial 7075 alloy (Reference 9) in Figure 6. It is apparent from this figure that MA87 exhibits behavior similar to that of 7075. A more practical way to look at the data is presented in Figure 7. It shows a representation of a 3-inch-thick product with hardness plotted at the surface and the midplane. As has been pointed out previously, the fast quench can represent the condition at the surface of such a product while the slow quench represents that of the midplane when the product is

TABLE 3

EFFECT OF NATURAL AGING ON ARTIFICIAL AGING RESPONSE OF MA87

CONDITION*	HARDNESS, R_B FAST QUENCH	SLOW QUENCH
AQ	42	48
AQ + NA	78	72
AQ+NA+time at 250°F(FHR)		
1 minute	67	63
2 minutes	69	64
5 minutes	70	66
10 minutes	73	67
30 minutes	77	73
AQ+NA+30 min @250°F(FHR) + 1410 minutes @ 250°F(SHR)	89	83
AQ + 1440 minutes @ 250°F(SHR)	88	81
AQ + 1440 minutes @ 250°F(FHR)	90	84
AQ + NA + 1440 minutes @ 250°F(SHR)	89	82
AQ + NA + 1440 minutes @ 250°F(FHR)	89	82

* AQ: as-quenched
NA: natural aged six days at room temperature
FHR: fast heating rate
SHR: slow heating rate

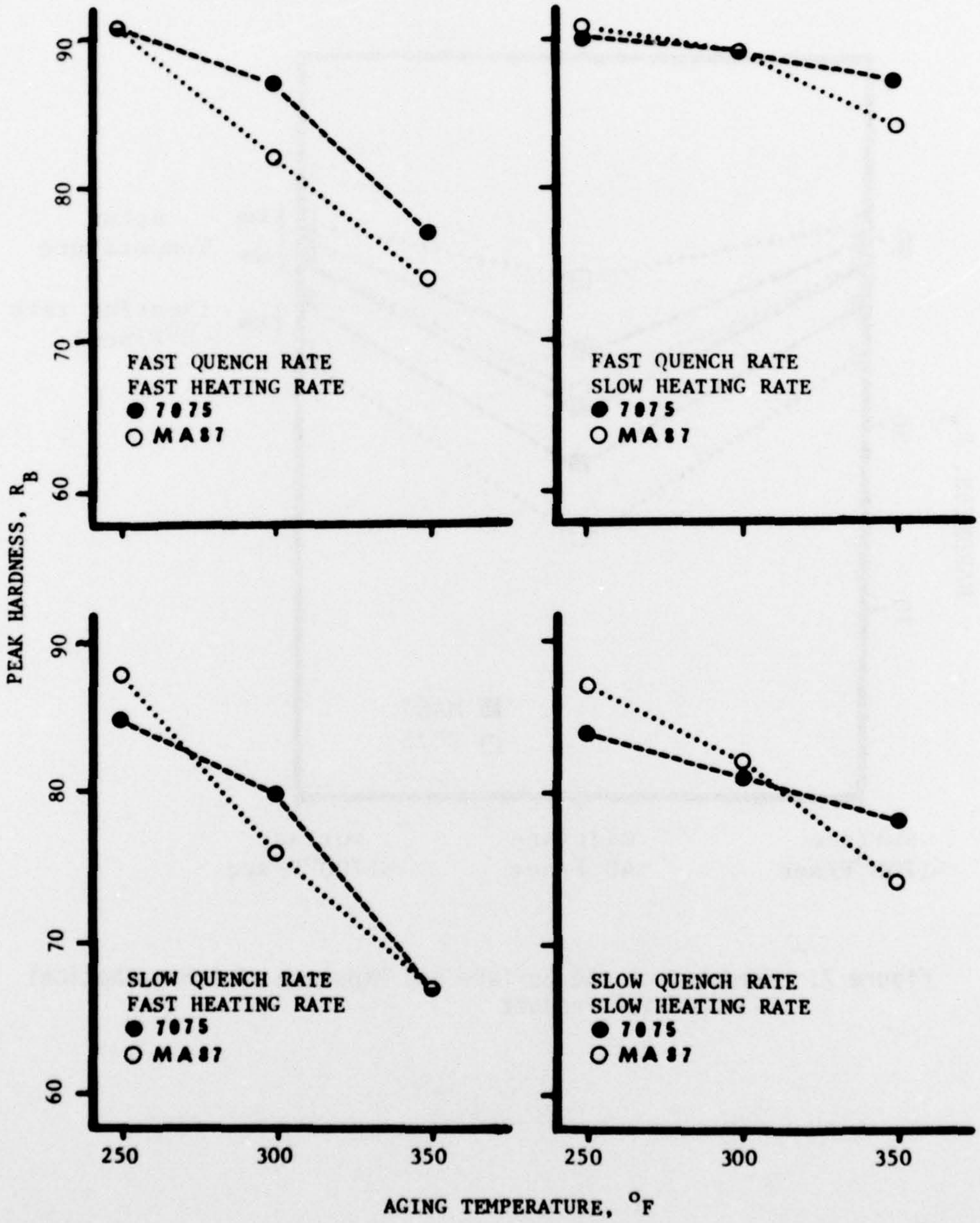


Figure 6. Peak Hardness vs. Aging Temperature for Various Quench Rate-Heating Rate Combinations

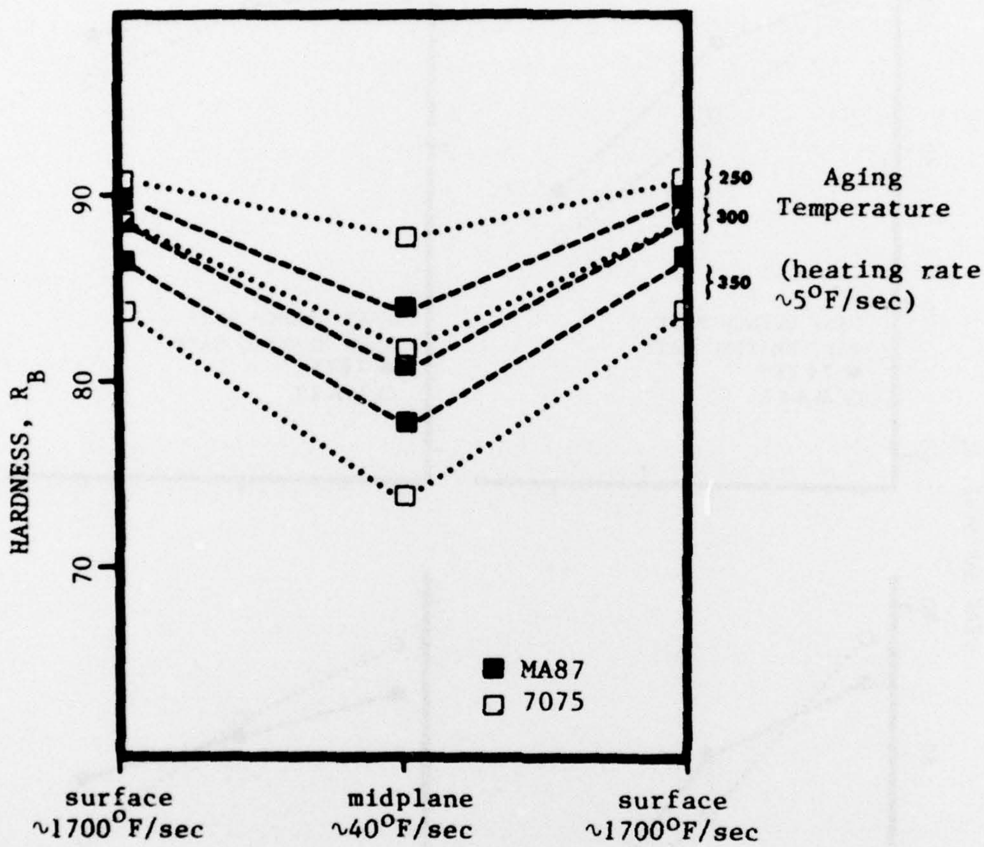
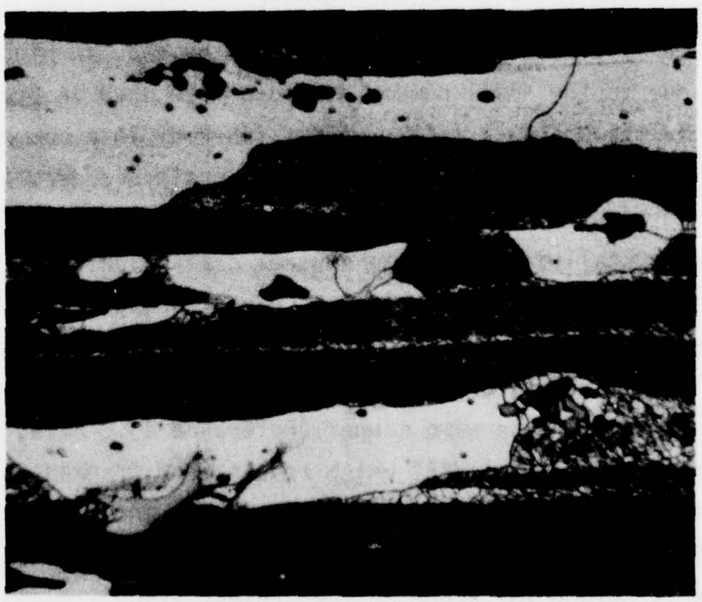


Figure 7. Hardness at the Surface and Midplane of a Hypothetical 3-Inch-Thick Product

quenched into 72°F water. In this case a measure of the quench sensitivity can be taken as the difference between the surface and midplane hardnesses. This hardness difference is approximately six R_B units for every alloy-temperature combination. The similarity of the behavior of MA87 to that of 7075 is attributable for the most part to the same phenomenon which causes a loss of strength in 7075, i.e., a reduction in the amount of solute (Mg, Zn, Cu) available for precipitation during aging due to heterogeneous nucleation and precipitation during the quench.

The absolute hardnesses represented in Figure 7 do vary with aging temperature. Note that at 250°F the 7075 alloy is harder than MA87; at 350°F, the trend is reversed with MA87 being harder. At 300°F there is little difference between the alloys. It is reasonable to believe that the differences that occur are due to differences in chemistry or microstructure, i.e., those factors influencing diffusion rates and thus precipitation kinetics. For example, MA87 has a finer grain structure than does normally processed ingot 7075 as can be seen in Figure 8. This is primarily due to the small powder particle size used in producing MA87 contrasted with the coarse grain structure inherent in a commercial ingot product. This fineness in MA87 provides more grain boundary area to promote heterogeneous nucleation. In addition, chromium is added to 7075 to suppress recrystallization and to control grain size; it has been demonstrated (References 10 and 11) that Cr-containing noncoherent dispersoid particles provide nucleation sites during quenching. Similarly, MA87 does contain 0.4% cobalt which forms the spherical noncoherent intermetallic Co_2Al_9 which may act in the same manner (Reference 4). Also, oxide particles are present in the MA87 which result from fragmentation of the oxide films that form during solidification of the individual powder particles. Determination of the magnitude of the contribution of each of these factors is beyond the scope of the present study.

(A)



(B)

Figure 8. Comparison of Microstructure of Powder and Commercially Processed Material. (A) Shows Fine Grain Structure of MA87 Forgings, which Is Due to the Small Powder Particle Size Used in Producing the P/M Product. (B) The Typically Coarse Grain Structure of 7075 Produced in a Commercially Processed Ingot Product (Echant-Boiling 1:1 HNO_3)

Figures 9 and 10 show the results of electron microscopic examination of MA87 and 7075. Figure 9 shows MA87 in the as-quenched condition. The predominant features of the microstructure are the spherical Co_2Al_9 particles and the finer oxide particles that appear as either clusters or as stringer-like dispersions. Figure 10a shows the grain boundary precipitation that occurs when a fast quench-slow heat specimen is aged at 350°F. Figure 10b indicates the grain boundary precipitate is more abundant and the precipitate-free zone (PFZ) adjacent to the grain boundary is wider than that for the fast quench condition. The PFZ indicates a region of solute depletion. The presence of a PFZ at Co_2Al_9 particles in the slow quench condition also show evidence of minor solute depletion adjacent to them. In Figures 10c and 10d, fast and slow quench conditions of the 7075 alloy are offered for comparison. Regions containing the Cr-containing dispersoids show solute depletion. Grain-boundary precipitation and accompanying PFZ's are also evident. Although not shown in any of the photomicrographs there was some evidence that the oxide may play a role in the solute depletion of the matrix. This is consistent with the observations of Cebulak and Lyle (Reference 12) who reported that there was evidence that oxide particles serve as nuclei for precipitation of $MgZn_2$ and that the interparticle spaces within clusters sometimes appear to be precipitate free in materials aged at 325°F.

These results for MA87 are consistent with those reported by Cebulak and Truax (Reference 13) on similar powder alloys. In their work they report "quench sensitivity is related to the amount of "M" phase $Mg(Zn, Cu, Al)_2$ which precipitates during the quench." Thus, it appears that the quench sensitivity of MA87 may be explained in terms of solute depletion due to nucleation at noncoherent interfaces during the quench from the solution heat treatment temperature.

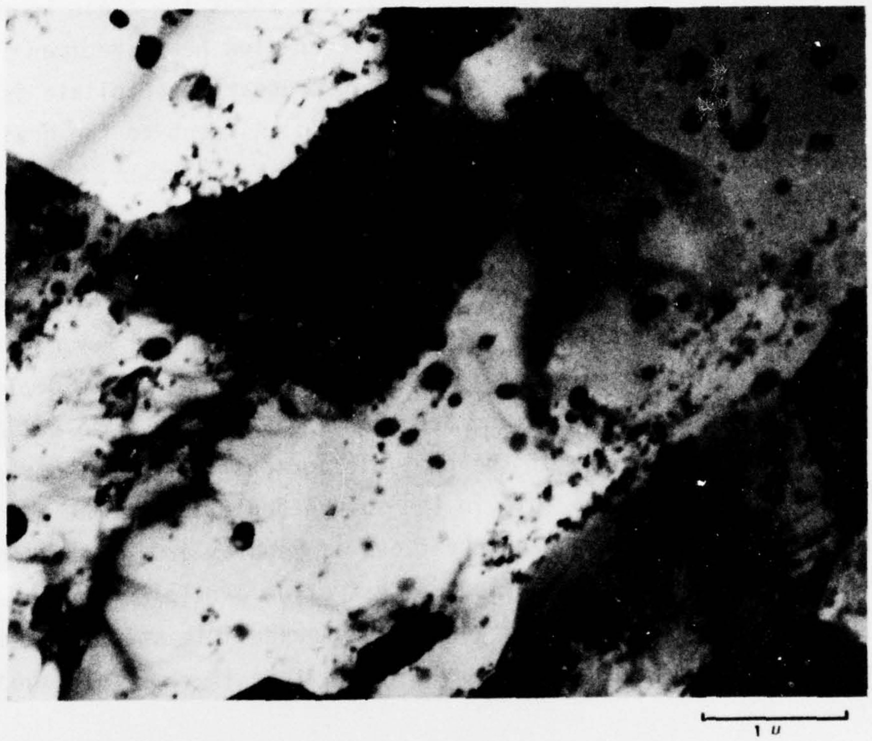


Figure 9. As-Quenched Microstructures of MA87. The Fine Particles Are Oxide and the Coarser Spherical Particles Are Co_2Al_9

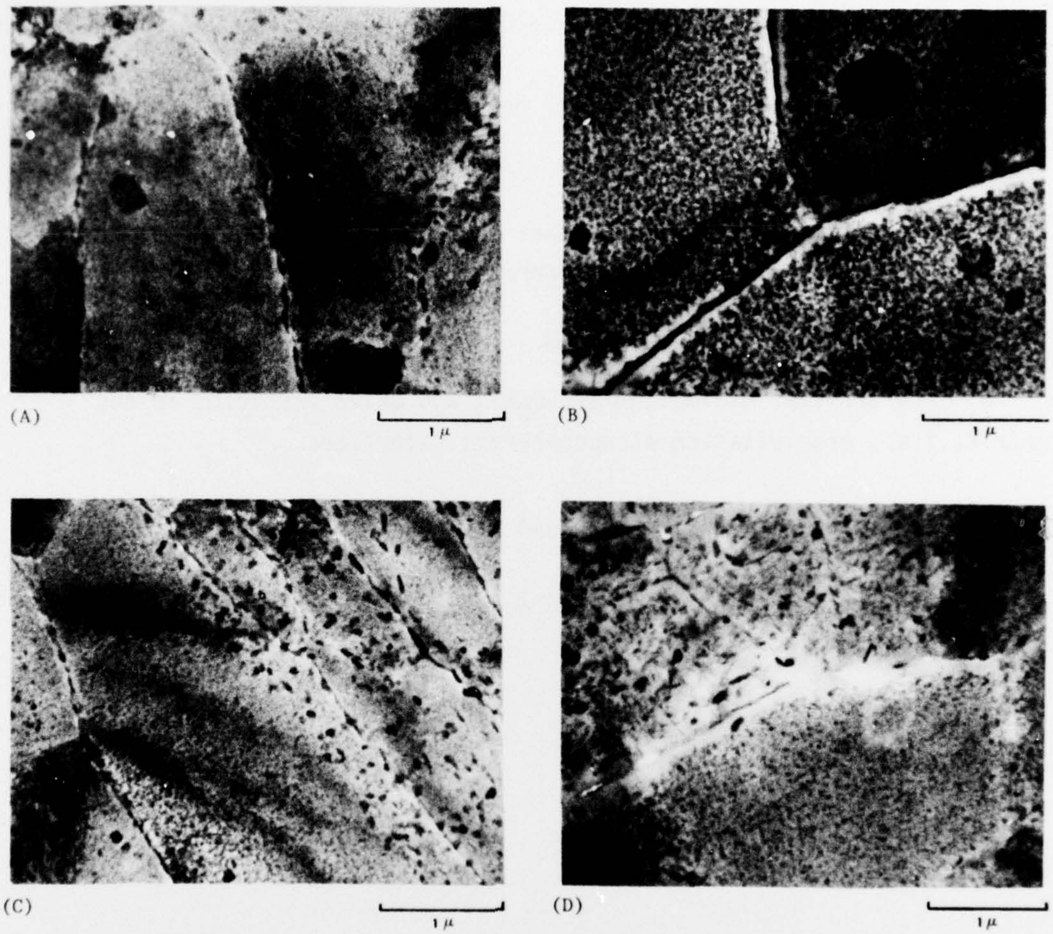


Figure 10. Variation in Age-Hardened Microstructure for Selected Quench and Heating Rate Combinations. (A) MA87; Fast Quench-Slow Heating, 350°F-5°Hours; (B) MA87; Slow Quench-Fast Heating, 350°F-50 Hours; (C) 7075; Fast Quench-Slow Age, 350°F-20 Hours; and, (D) 7075; Slow Quench-Fast Age, 350°F-48 Hours

SECTION IV

CONCLUSIONS

1. From a hardness standpoint, MA87 and 7075 have similar quench sensitivity behavior.

2. Quench sensitivity in MA87 appears to be due to solute depletion in the matrix through two principal means:

   a. grain boundary precipitation

   b. preferential nucleation at Co_2Al_9 and oxide particles in the matrix, i.e., precipitation at noncoherent interfaces.

3. MA87 exhibits a reversion response after natural aging.

4. Natural aging appears to provide no advantages for MA87 aged at 250°F, in terms of hardness.

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